

U.S. DEPARTMENT OF COMMERCE PATENT AND AND

TRANMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

INTERNATIONAL APPLICATION NO. PCT/IB99/01700

INTERNATIONAL FILING DATE 18 October 1999

ATTORNEY'S DOCKET NUMBER

HINZE 1

U.S. APPLICATION NO. (If known.see 37 CFR 1.5)

PRIORITY CLAIMED

23 October 1998

TITLE OF INVENTION

BACTERICIDAL TREATMENT OF FOOD STORAGE CONTAINERS BY USING ELECTROCHEMICALLY ACTIVATED BACTERICIDAL AQUEOUS SOLUTION

APPLICANT(S) FOR DO/EO/US

Gilbert HINZE

CCTER.

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Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

- 1. [X] This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
- 2. [] This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
- 3. [X] This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
- 4. [X] The US has been elected in a Demand by the expiration of 19 months from the priority date (PCT Article 31).
- 5. [X] A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. [] is attached hereto (required only if not transmitted by the International Bureau).
 - b. [X] has been communicated by the International Bureau.
 - c. [] is not required, as the application was filed in the United States Receiving Office (RO/US).
- 6. [] An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
- 7. [X] Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. [] are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. [] have been communicated by the International Bureau.
 - c. [] have not been made; however, the time limit for making such amendments has NOT expired.
 - d. [X] have not been made and will not be made.
- 8. [] An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
- 9. [] An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
- 10. [] An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11, to 16, below concern document(s) or information included:

- 11. [] An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
- 12. [] An Assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
- 13. [X] A FIRST preliminary amendment.
 - [] A SECOND or SUBSEQUENT preliminary amendment.
- 14. [] A substitute specification.
- 15. [] A change of power of attorney and/or address letter.
- 16. [X] Other items or information:
 - [X] Courtesy copy of the International Application as filed.
 - [X] Courtesy copy of the first page of the International Publication (WO 00/24275).
 - [X] Courtesy copy of the International Preliminary Examination Report. There were no annexes
 - [X] Courtesy Copy of the International Search Report.

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Gilbert HINZE) Art Unit:
IA No.: PCT/IB99/01700	
IA Filed: 18 October 1999) Washington, D.C.
U.S. App. No.: (Not Yet Assigned)	April 23, 2001
National Filing Date: (Not Yet Received))
For: BACTERICIDAL TREATMENT) Docket No.: HINZE

PRELIMINARY AMENDMENT

Honorable Commissioner for Patents and Trademarks Washington, D.C. 20231

Sir:

Contemporaneous with the filing of this case and prior to calculation of the filing fee, kindly amend as follows:

IN THE SPECIFICATION

 $\label{eq:After the title please insert the following paragraph:} After the title please insert the following$

REFERENCE TO RELATED APPLICATIONS

The present application is the national stage under 35 U.S.C. 371 of international application PCT/IB99/01700, filed 18 October 1999 which designated the United States, and which international application was published under PCT Article 21(2) in the English language.

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REMARKS

The above amendment to the specification is being made to insert reference to the PCT application of which the present case is a U.S. national stage.

 $\label{eq:Favorable} \mbox{Favorable consideration and allowance are earnestly solicited.}$

Respectfully submitted,

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PCT/IB99/01700

BACTERICIDAL TREATMENT OF FOOD STORAGE CONTAINERS BY USING ELECTROCHEMICALLY ACTIVATED BACTERICIDAL AQUEOUS SOLUTION

TECHNICAL FIELD

This invention relates to bactericidal treatment of bio-film in food storage containers. More particularly, the invention relates to bactericidal treatment of bio-film in bulk food storage containers used for fresh produce.

BACKGROUND ART

One of the problems with all fresh produce is their perishable nature and thus their limited shelf life. This is largely due to bacterial contamination and putrefactive enzyme production by the bacteria. A primary source of bacterial contamination is the bacterial bio-film that exists on the inside of bulk storage containers, such as those used on fishing trawlers.

For purposes of this specification, the term "fresh produce" shall be interpreted so as to include fresh foodstuff such as fish, chicken, meat, meat carcasses, processed meat products, processed chicken products, processed fish products and the like.

The use of bulk food storage containers for fresh fish such as those on fishing boats and trawlers, often constituted by the hulls themselves, travelling out to sea for lengthy periods on their fishing trips, is well known. As the fish are caught they are stores typically in crushed ice in the storage containers and hulls of trawlers and boats. Once

sufficient fish have been caught, the trawlers return to harbour where the fish are off-loaded and processed. In many cases much of the preliminary processing, such as "gutting", is done on board out at sea.

Through the storage of freshly caught fish in these hulls, the fish are exposed to bacterial contamination from the bio-film and the gut residue, thereby reducing the shelf life of the fish.

The use of other bulk food storage containers, such as those used on road and rail transporters for fresh produce such as cattle and sheep carcasses, is similarly well known. In these containers the bio-film on the inside of the storage hulls originates also from blood and gut residue as well as previously contaminated carcasses.

For purposes of this specification, the term "bulk food storage containers" shall be interpreted so as to include containers used for fresh produce such as fresh fish on trawlers, sheep and cattle carcasses on road transporters, rail transporters and the like, and associated terms shall be interpreted so as to have cognate meanings.

Further and for purposes of this specification, the term "transporter" shall be interpreted so as to include fishing ships and trawlers, bulk road

and rail transporters for fresh produce and the like.

OBJECTIVES OF THE INVENTION

It is accordingly an object of the invention to increase the shelf life of fresh produce in bulk food storage containers by overcoming or at least minimising the above disadvantage.

DISCLOSURE OF INVENTION

According to a first aspect of the invention there is provided a method for bactericidal treatment of bulk food storage containers for fresh produce, the method including the step of treating a container with electrochemically activated, bactericidal aqueous solution.

According to a second aspect of the invention there is provided fresh produce, characterised in that it has been treated with electrochemically activated, bactericidal aqueous solution during storage in a bulk food storage container.

According to a third aspect of the invention there is provided a bulk food storage facility, including a bulk food storage container, for fresh produce, the facility being characterised in that it includes means for producing electrochemically activated, bactericidal aqueous solution for

treating an internal surface of the container.

According to a fourth aspect of the invention there is provided a transporter, having a bulk food storage container for transporting fresh produce, the transporter being characterised in that it is provided with means for producing electrochemically activated, bactericidal aqueous solution.

The method may include the step of packing the fresh produce in ice in the container, the ice being characterised in that it is made from an electrochemically activated, bactericidal aqueous solution.

The transporter may be provided with means for providing the aqueous solution in iced form.

The electrochemically activated, bactericidal aqueous solution may be selected from the group consisting of mixed oxidant, anion-containing aqueous solution and mixed reductant, cation-containing aqueous solution.

The electrochemically activated, bactericidal aqueous solution may be prepared by means of electrolysis of an aqueous solution of a salt. The

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salt may be sodium chloride. In particular, it may be non-iodated sodium chloride or potassium chloride.

The anion-containing solution and the cation-containing solution may be produced by an electrochemical reactor or so-called electrolysis device, having a through flow electrochemical cell with two co-axial cylindrical electrodes, with a co-axial diaphragm between them so as to separate an annular inter-electrode space into a catalytic and an analytic chamber. The anion-containing solution is referred to hereinafter for brevity as the "anolyte solution" or "anolyte" and the cation-containing solution is referred to hereinafter for brevity as the "catholyte solution" or "catholyte".

The electrochemically activated, bactericidal aqueous solution may be produced from an about 3 to 10% aqueous NaCl solution, electrolysed to produce mixed reductant and mixed oxidant species. These mixed oxidant and reductant species may be labile and after about 96 hours, the various radical species may disappear with relatively no residues being produced.

The analyte solution may have a redox potential of about between $+450 \, \mathrm{mV}$ and $+1200 \, \mathrm{mV}$ and a pH of between 2 and 9. The analyte

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solution may include mixed oxidant species such as CIO; CIO; HCIO; OH; HO $_2$; H $_2$ O $_2$; O $_3$; S $_2$ O $_8$ ² and CI $_2$ O $_6$ ².

These species have been found to have a synergistic anti-bacterial and/or anti-viral effect, which is generally stronger than that of chemical bactericides and has been found to be particularly effective against viral organisms and spore and cyst forming bacteria.

The catholyte solution generally may have a pH of between about 12 and 13 and a redox potential of between about -850mV and -900mV. The catholyte solution may include mixed reductant species such as OH; H_3 ; O_2 ; H_2 ; HO_2 ; HO_2 ; and O_2 .

According to a fifth aspect of the invention there is provided equipment for use in a method for bactericidal treatment of bulk storage containers for fresh produce, the apparatus including an electrolysis device, having a through flow electrochemical cell with two co-axial cylindrical electrodes, with a co-axial diaphragm between the two electrodes so as to separate an annular inter-electrode space into a catalytic and an analytic chamber.

Both the physical characteristics of the anolyte and the catholyte, such as pH and redox potential, are adjustable so as to be suitable for a

particular application, such as type of produce, the atmospheric conditions in the container and the like.

BEST MODES FOR CARRYING OUT THE INVENTION

A preferred embodiment of the invention will now be described as a non-.... limiting example only.

Example 1:

By using anolyte, it is envisaged that one can achieve an increased shelf life for fish of up to 3 to 9 days. The proposed application of anolyte is as follows:

- (a) As ice for storage purposes; and
- (b) As a method of eliminating the bio-film on the inside surfaces of bulk storage containers such as those on fishing trawlers and boats.

1.1 <u>lce</u>

By using anolyte in the form of ice in the storage of fish, the bacterial contamination in the ice is eliminated as well as the contamination of the packed fish. As the ice melts, the anolyte is released to destroy the bacteria.

It is envisaged that the anolyte can be iced either as a concentrate, or in a diluted state with water, varying in dilution from 50% to as low as 20% dilution. The dilution would depend largely upon the contaminated state of the water used in the ice. Some trawlers, for example, use seawater in their ice. Seawater by nature is very contaminated.

The type of analyte to be used in the ice is :

pH - ± 7.5;

Amps - 12 - 13 amps (24 volt);

ORP - \pm 450 mV; and

Pressure - 0.5 bar (720ml/hr - production rate)

1.2 Bio Film:

Through applying anolyte as a fog within an empty storage container of a trawler, one could eliminate the bio-film and thus the risk of re-contamination of the fish during subsequent use.

The elimination of this bio-film will generally take place between fishing trips, while the trawler is in the harbour with its storage containers empty.

It is envisaged that various methods of application such as fogging could be applied, as long as the droplet size of the fogged analyte is small (around 4 to 12 micrometers) and the contact time is sufficient. Depending upon the extent of the bio-film, a number of fogging sessions could be required.

Fogging time will also depend upon the size and volume of the container and the output of the fogging apparatus. Generally, one will fog until a thick fog has formed in the closed container and the walls of the container have been sufficiently wet by the anolyte fog so that droplets begin to form and run off (run-off stage). The container would then be allowed to dry before being fogged again.

It is envisaged that the type of anolyte to be used could be:

PH - ± 6.5;

Amps - 12 - 13 amps (24 volts);

ORP - ± 750mV; and

Pressure - 0.5 bar (± 750 ml/hr - output)

It is envisaged that it could be advantageous also to use anolyte as a general disinfectant in the processing and putting of the fish, of both the process facilities and equipment and the product itself.

Anolyte has very limited residue and thus an advantageous over the other disinfectants on the market that are generally chemically based.

Example 2:

Multiple fogging cycles were used so as to determine the efficacy thereof on the total bacterial surface loads in a series of chillers over a 42 hour chilling period.

Samples 1, 2 and 3 were carcasses fogged separately in chillers with 30 minute intervals. Samples 4 and 5 were carcasses sampled in operating chillers. Foggers were put on the floor of the chillers and carcasses were therefore not fogged directly. 3 x sampling was conducted 42 hours after the previous fogging on all samples so as to establish whether there would be an increase in bacterial loads over the 42 hours prior to de-boning.

Multiple fogging in areas where the fog is not mechanically removed from

the room during the fogging process is highly effective in reducing total counts.

Fogging in operating chillers is not effective.

Throughout the trial Coliform counts were low, most probably due to carcass washing and results therefore were not given.

Example 3:

Enclosed volumes containing diverse equipment, including 2 tables and a scale, were fogged so as to determine the microcidal effect of analyte on the enclosure surfaces and the enclosed equipment. The results are shown in the accompanying tables.

Example 4:

Cattle carcasses were treated at the Agricultural Research Council Unit, Irene, Gauteng, South Africa.

The analyte used was generated under and with the following characteristics:

Current: 10 Ampere; Voltage: 24 Volt

ORP : +762 mV: TDS: 6.04 q/ℓ

PH : 6,8

The chiller treated had volume (space) for about 16 carcasses. The fogging process consisted of 3 cycles of 20 minutes each, with 10 minutes in between each cycle.

Samples were taken from the neck area, the breast area, the back area and the hindquarter area.

Samples were taken of all micro-organisms by means of total plate count (Redoc plates), total plate counts (petri film) and Coliforms (petri film).

The results are shown in the accompanying tables.

Example 5:

A number of 800 lamb carcasses were subjected to tests, 400 being fogged with analyte and 400 being used as the control group. Samples

were taken before treatment, after a second cycle and a fourth cycle, while the control group was sampled before and after 24 hours of chilling.

Additional samples were taken from both the treated and the control group for measuring TPC only.

The results are shown in the accompanying tables.

It will be appreciated that many variations in detail are possible without departing from the scope and/or spirit of the invention as claimed in the claims hereinafter.

Example 2: (New Style Pork)

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Objective:

To determine the effect of multiple fogging on the total bacterial surface loads over a 42 hour chilling period.

	Colony Forming us Fogging frequ		ming units / 10a	em	Ei	ficacy	Anolyte production Parameters	
1	No.	Control	1x	2x	3x(+36h)	% Red.	Log red.	Amp pH
	169	TNTC	12	5	7	99.9	-6	
	236	TNTC	360	3	1	99.9	-6	9 6.66
	168	600	6	3	1	99.9	-2	1
	246	110	120	2	1	99.9	-2	24V
	170	13	3	70	0	99.9	-1	1.0 Bar
	99.7	-3.4						
2	189	12	7	1	30	0	0	
	203	100	8	15	0	99.9	-2	12 6.8
	200	140	13	1	0	99.9	-2	
	192	80	2	35	2	97.5	-1	24V
	232	200	6	80	0	99.9	-2	1.0 Bar
	79.4	-1.4						
3	193	TNTC	100	3	2	99.9	-6	
	177	2	30	20	10	0	0	10 5.0
	178	110	70	50	24	78.2	-1	
	179	13	10	6	1	92.3	-1	24V
	180	110	22	11	2	98.2	-2	1.0 Bar
	73.7	-1.8						
4	200	1	4	-	2	0	0	
	211	8	100	-	50	0	0	11 5.0
	1304	5	1	-	0	99	0	
	194	5	8	-	2	40	0	24V
	245	3	4	-	1	33	0	1.0 Bar
	34.4	0						
5	58	1	210	-	TNTC	0	0	
	106	160	120	-	12	92	-2	9 6.6
Г	68	6	4	-	3	50	-1	
	94	2	60	-	4	0	0	24V
	32	110	90	-	70	27	-1	1.0 Bar
	33.8	.08						

Comments

2 and 3 were fogged separately in chillers with 30 minutes intervals.
 Chillers were not in operation. After fogging, carcasses were returned to original chillers.

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Example 3: Microcidal Effect of Anolyte on Surfaces and Equipment

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The test conditions were as follows: ANOLYTE:

10 Amp 24 volt

+ 762 mV ORP 6.04g/l TDS 6.8pH

Chiller capacity: No. of carcass in chiller

Fogging:

Neck area, breast are, back area, hindquarter area Total plate count (Rodac plates)
Total plate counts (petri film)
Coliforns (petri film) 3 x 20 min (10 min rest in between) 9

Direct fogging in chiller with interrupted air circulation during the fogging process

Trial Carcasses

Ą.

Mico-organisms: Samples taken:

			The same of the sa		
		CFU/10e	$\mathrm{CFU/10cm}^2$ – Total Aerobic Count Petri film	etri film	
	Before	1 x	2 x	3 x	4 x
V1 Right	168	38	22	18	∞ ¦
Left V2 Right	58	33		2 18	4
Left V3 Right	330	69	25	0	106 .
Left V4 Right	156	56 56	58	8 20	154
V5 Right	220	72	78	19	130
Left V6 Right	175	112	110	16	160
Left Mean R	185	62	59	11	þ6
Ø, Dacresse		-62	89-	-94	-51

Comments:

The fourth Swab was on the side of the tricops cut where all carcasses had been pushed by hand and were therefore more contaminated than adjoining surfaces.

Coliform counts were negligible on all carcasses All swabs were incubated at 37° C for 48 hours

Negative Control: Indirect fogging of carcasses that were present in the chiller, during the time of the experiment. Only final carcass counts on similar locations as the trials were taken.

CFU/10cm²	17	24	5	55	89	14	3	27
#		2	80	4	5	9	7	Mean

Example 5:

Woolworths Trial 800 lamb carcasses

Results:

Treatment with Analyte

Carcase #	Befo	re treatme	nt	Af	ter 2 nd fogs	ing	A	ter 4th fogg	
	TPC	Coliform	E.coli	TPC	Coliform	E.coli	TPC	Coliform	E.coli
B33537	180	0	0	-	-	-	0	1	0
B28392	28	0	0	-	-	-	0	0	0
B29673	27	0	1	9	0	0	5	0	0
B30680	19	0	0	0	0	0	0	0	0
B32522	190	14	0	-	-	-	0	0	0
B29535	3	0	0	4	0	0	0	0	0
B28258	18	0	2	5	0	0	1	0	0
B29602	600	0	0	-	-	-	3	0	0
B29505	23	0	0	8	0	0	2	0	0
B28659	25	0	0	-	-	-	0	0	0
Total	1113	14	3	26	0	0	11	1	0
	111.3	1.4	0.3	5.2	0	0	1.1	0.1	0
20cm ²									

Control group:

Carcase #		Before Chilling	After chilling 24 hi		
	TPC Coliform		E.coli	TPC	
C32592	21	6	0	17	
C28363	38	0	0	27	
C29469	11	23	0	15	
C32540	84	2	0	166	
C28309	17	0	0	48	
C29137	614	0	0	228	
C28588	9	0	0	123	
C33039	0	0	0	0	
C28333	38	1	1	179	
C30032	2	0	0	0	
Total	834	834 32		803	
Mean/ 20cm ²	83	3.2	0	80	

Further swabs were taken on the shoulder of 5 chilled and fogged carcases (after the 4th fogging).

Carcase #	TPC
4BS1	1
4BS2	3
4BS3	0
4BS4	6
4BS5	6
Total	16
Mean/20cm ²	3.2

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CLAIMS

- A method for bactericidal treatment of bulk food storage containers for fresh produce, the method including the step of treating a container with electrochemically activated, bactericidal aqueous solution.
- Fresh produce, characterised in that it has been treated with electrochemically activated, bactericidal aqueous solution during storage in a bulk food storage container.
- 3. A bulk food storage facility, including a bulk food storage container for fresh produce, the facility being characterised in that it includes means for producing electrochemically activated, bactericidal aqueous solution for treating an internal surface of the container.
- 4. A transporter, having a bulk food storage container for transporting fresh produce, the transported being characterised in that it is provided with means for producing electrochemically activated, bactericidal aqueous solution.

- 5. A method as claimed in claim 1, including the step of packing the fresh produce in ice in the container, the ice being characterised in that it was made from the electrochemically activated, bactericidal aqueous solution.
- A bulk food storage facility as claimed I claim 3, characterised in being provided with means for producing the aqueous solution in iced form.
- A method as claimed in claim 1, wherein the aqueous solution is selected from a group consisting of mixed oxidant, anioncontaining solution and mixed reductant, cation-containing solution.
- A method as claimed in claim 7 wherein the solution is produced from an about 3 to 10% aqueous salt solution, electrolysed to produce mixed reductant and mixed oxidant species.
- A method as claimed in claim 8 wherein the salt solution is sodium chloride or potassium chloride solution.

- 10. A method as claimed in claim 1 wherein the electrochemically activated, bactericidal aqueous solution is anion-containing solution is produced by an electrolysis device, having a through flow electrochemical cell with two co-axial cylindrical electrodes, with a co-axial diaphragm between the two electrodes so as to separate an annular inter-electrode space into a catalytic and an analytic chamber.
- 11. A method as claimed in claim 8 wherein the species are labile and wherein they, after about 96 hours, disappear with relatively no residues being produced.
- 12. A method as claimed in claim 7 wherein the anion-containing solution has a redox potential of between about +450 mV and + 1200 mV and a pH of between about 2 and 9.
- 13. A method as claimed in claim 7 wherein the anion-containing solution includes mixed oxidant species selected from the group consisting of CIO; CIO; HCIO; OH; HO₂; H₂O₂; O₃; S₂O₈² and CI₂O₆²

- 14. A method as claimed in claim 7 wherein the cation-containing solution has a pH of between about 7 and 13 and a redox potential of between about -200 mV and -900 mV.
- 15. A method as claimed in claim 7 wherein the cation-containing solution includes mixed reductant species selected from the group consisting of OH'; H₃'; O₂; H₂; HO₂; HO₂ and O₂.
- 16. A method as claimed in claim 1 wherein the physical characteristics of the anion-containing and the cation-containing solution are adjustable so as to be suitable for the particular application.

ABSTRACT

The invention relates to a method for bactericidal treatment of bulk food storage containers for fresh produce, the method including the step of treating a container with electrochemically activated, bactericidal aqueous solution.

[X] Original [] Substitute [] Supplemental

Combined Declaration for Patent Application and Power of Attorney

Page 1 of 2 Pages

As a below-named inventor, I hereby declare that:

Atty. Docket: HINZE 1

The undersigned hereby authorizes the U.S. Attorneys or Agents appointed herein to accept and follow instructions from D.M. KISCH RNC, as to any action to be taken in the U.S. Patent and Trademark Office regarding this application without direct communication between the U.S. Attorneys or Agents and the undersigned. In the event of e change of the persons from whom instructions may be taken, the U.S. Attorneys or Agents appointed herein will be so notified by the undersigned.

As a named inventor, I hereby appoint the following registered practitioners to prosecute this application and to transact all

All of the practitioners associated with Customer Number 901444

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Page 2 of 2 Pages			Docket: HINZE 1
Title: BACTERICIDAL TREATMENT OF FOO		BY USING EL	ECTROCHEMICALLY
ACTIVATED BACTERICIDAL AQUEOUS SOLUTION			
U.S. Application filed April 23, 2001 PCT Application filed October 18, 1999	, Serial No.	700	
PC i Application filed October 18, 1999	, Serial No. FC (/ID99/V)	700	
I hereby further declare that all statements made herein			
and belief are believed to be true; and that these stateme			
so made are punishable by fine or imprisonment, or jeopardize the validity of the application or any patent is	soun, under is 0.5.C. givel ar	ia that such will	ui tause statements may
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